THE EFFECT OF PRIMER APPLICATION MODIFICATIONS ON THE BOND STRENGTH OF 4TH GENERATION ADHESIVE BONDING AGENTS

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By

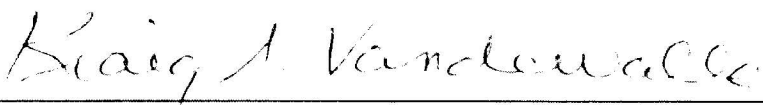Harold S. Zald BS, DDS

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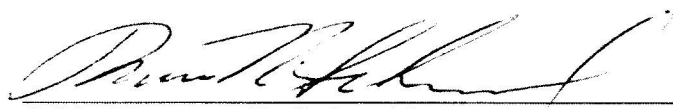
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DEDICATION

This thesis is dedicated to my wife, Dianne. She has been immensely supportive and always enthusiastic as I took on this unusual, late-in-life transition from civilian private practice to serving as a Naval Officer.

In spite of the uncertainties of a long recruiting and commissioning process, followed by extended absences during training and operational assignments on both coasts, Dianne has stayed positive throughout, even while working through the difficult process of leaving behind a full and comfortable life in Michigan, with the loss of close proximity to long time friends, colleagues and family. Dianne has gracefully adapted during these disruptive years, and with her sense of adventure, actually embraced these tumultuous changes as few other people I know could……or would.

Subsequently, she has shown me nothing but encouragement, as I undertook the time-consuming clinical, didactic and research challenges presented by the USAF AEGD-2 program, in order to pursue my career dream of becoming a "master generalist", a Navy "Comp" Dentist, as well as an Air Force "A".

Thanks for your enthusiastic love and support, Dianne, as we leave Texas and start a new "Pacific" chapter in our adventure together……

"Hu guiaya hao!"

ACKNOWLEDGEMENTS

ABSTRACT

Objectives: The purpose of this study was to evaluate the effect of variations in application technique of the primer component of three-step, etch-and-rinse adhesive agents on the shear bond strength of composite resin to dentin.

Methods: The coronal enamel of 120 extracted human third molars was removed with a low-speed saw. The teeth were mounted in PVC pipe with dental stone, and randomly divided into 12 groups of 10 teeth each. The flat dentin surfaces were bonded using the manufacturer's directions (MD) for Adper Scotchbond Multipurpose (3M/ESPE, St. Paul, MN) and Optibond FL (Kerr, Orange, CA). The variation from MD for each primer agent were: 1) application method (passive or active), or 2) application time (MD, MD+10 seconds, or MD+20 seconds). The adhesives were light cured and the specimens were placed in a jig (Ultradent, South Jordan, UT). Filtek Z250 (3M/ESPE, St.Paul,MN) composite was light cured in 2-mm increments to 3-4 mm in height. Specimens were stored for 24 hours in 37°C distilled water before testing in a universal testing machine (Instron, Norwood, MA). A mean and standard deviation were determined per group. Data were analyzed with ANOVA/Tukey's.

Results: With Optibond FL, a two-way ANOVA found no significant difference between groups based on application time or method with no significant interaction ($p>0.05$). With Adper Scotchbond Multipurpose, a two-way ANOVA found no significant differences based on application time or method ($p>0.05$), but there was a significant interaction ($p=0.002$). One-way ANOVAs and Tukey's t-tests found a significant difference between groups ($p<0.001$). See table.

Conclusions: With Optibond FL, there was no significant difference in shear bond strength to dentin based on application time, or active versus passive application of the primer. However, active application of the primer of Adper Scotchbond MP at the manufacturer's recommended application time increased the bond strength compared to longer application times or passive application.

Application Time (seconds) Manufacturer's Directions (MD)	Mean Shear Bond Strength MPa (st dev)			
	Scotchbond MP		Optibond FL	
	Active	Passive	Active	Passive
MD	17.46 (6.33) Aa	8.60 (4.66) Ba	14.27 (4.91)	9.92 (6.20)
MD + 10 secs	11.19 (6.48) Ab	11.61 (2.91) Aa	10.20 (5.39)	7.80 (4.25)
MD + 20 secs	9.21 (3.36) Ab	12.57 (6.53) Aa	8.50 (3.64)	9.33 (4.49)
	Groups with the same lower case letter per column or upper case letter by row are not significantly different (p>0.05)			

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I. BACKGROUND AND LITERATURE REVIEW

A. Background

1. Acid Etching in Dentistry.

Buonocore demonstrated in 1955 that the preparation of dental enamel with an acid-etching technique enhanced the adhesion of acrylic filling materials to prepared teeth (Buonocore, 1955). Since that time, acid-etch preparation of teeth, along with the development of newer adhesive resin systems, composite resin materials, and ceramics, has stimulated an explosive expansion of esthetic restorative dentistry.

Over the last 30-35 years, the restorative systems marketed by various dental manufacturers have evolved dramatically with demonstrable advances in durability and esthetics. Complexity and time of application are a concern in clinical practice. Dental material manufacturers have responded by ultimately developing newer, simplified, adhesive resin systems (Perdigao and Swift, 2010).

2. Current State of Dental Bonding Technologies.

There are now seven generations of adhesives, with each generation reflecting an attempt to reduce procedure steps and time of application (Table 1).

Table 1. Overview of Dental Adhesive Systems

	First Step	Second Step	Third Step	Fourth Step	Example	Bond Strength
Fourth Generation	Conditioner/Etch Apply to tooth Rinse	Primer Apply to tooth	Adhesive Apply to tooth	Light Cure	ScotchBond Multipurpose Optibond FL	High
Fifth Generation	Conditioner/Etch Apply to tooth	Primer+Adhesive Apply to tooth	Light Cure		Optibond Solo Prime & Bond NT Single Bond One Step	Moderate to High
Sixth Generation	Acidified Primer Apply to tooth Rinse	Adhesive Apply to tooth	Light Cure		ClearFil SE	Moderate to High
Sixth Generation	Conditioner Primer Adhesive Single mix applicator blister package Apply to tooth	Light Cure			Prompt-L-Pop	Low to moderate
Seventh Generation	Conditioner Primer Adhesive Apply to tooth	Light Cure			iBond G Bond	Low

Fourth generation adhesives came on to the market in the early 1990's. These three-step, etch-and-rinse systems require the use of an etchant (32-35% phosphoric acid), a primer, and an adhesive. These adhesives exhibited high bond strengths, with 98-100% retention of Class V restorations at 3 years in clinical studies. Examples include Adper Scotchbond Multipurpose (3M-ESPE, St. Paul, MN) and Optibond FL (Kerr, Orange, CA) (Perdigao and Swift, 2010; Brucia, 2010; Summit and Robbins, 2006).

Fifth generation adhesives evolved in the late 1990's and represented attempts to simplify procedural complexity by reducing the number of bottles in the system. These adhesives are also etch-and-rinse systems, using a separate application of etch, but combine the primer and adhesive into one simplified bottle. They still involve a post-etch rinse step, and have proven to be more technique sensitive, as the level of dentin wetness proved important. They have a higher solvent to monomer ratio which can negatively affect the material over a period of time, as the solvent evaporates with each use of the bottle. In practice they were often applied too thinly and required application in multiple layers. They have been well studied under long term clinical conditions. Examples include Optibond Solo (Kerr), Single Bond (3M-ESPE), and Prime & Bond NT (Dentsply, Milford, DE) (Perdigao et.al; 2009; Brucia, 2010; Summit and Robbins, 2006).

Sixth generation adhesives were the first of the self-etching systems. The sixth generation materials consist of two types:

1) A "two step" with the first bottle containing a combined conditioner and primer that is mixed with a adhesive contained in a second, separate bottle and then applied. An example would be Clearfil SE Bond (Kuraray, New York, NY). They have demonstrated moderate-to-high bond strengths.

2) A "one step" acidified primer and adhesive which has the components that must be mixed together and is then applied in one step. An example is Prompt-L-Bond (3M ESPE). (Perdigao and Swift, 2010; Brucia, 2010; Summit and Robbins, 2006).

Seventh generation or one-step, self-etch adhesives were the most recently developed agents. They are marketed as "simplified" or "all-in-one" adhesives which combine acidified primers and an adhesive in one bottle. Seventh generation adhesives require no mixing and are applied directly from the container, which eliminates a clinical step. They have exhibited low bond strengths relative to earlier generations. Examples of this new generation of materials would include iBond (Heraeus Kulzer, South Bend, IN) and G-Bond (GC, Alsip, IL). (Perdigao 2010; Brucia, 2010; Summit and Robbins, 2006).

B.   Review of the Literature

Review of literature revealed numerous studies that focused on the fifth-, sixth- and seventh-generation adhesives and variations in their application technique. In addition, literature searches were conducted specifically concentrating on investigations of fourth-generation, three-step etch-and-rinse materials and how variations in their primer component application technique affected bond strengths.

Only one study investigated the relationship between active versus passive primer application and its affect on bond strength within the category of three-step, etch-and-rinse systems. The adhesive bonding systems compared in that study were Imperva Bond (Shofu, Menlo Park, CA) and Scotchbond Multipurpose (3M ESPE, St. Paul, MN). Two experiments were run simultaneously.

The first experiment measured the effect of primer application procedures. The dentin primer was just applied (inactive application) or was applied and agitated by a brush (active application) using a 30-second application time for Imperva Bond and 10 seconds for Scotchbond Multipurpose. An increase in bond strengths was found with active application compared to inactive, although this difference was not found to be statistically significant.

The second experiment measured the effect of air drying time after application of the primer according to the manufacturer's directions. The effect of air drying time was measured at 0, 5, 10, 20 and 30 seconds from 10 cm above the dentin surface. There was an optimal range of drying times for each restorative system. This study also reported that the fourth-generation adhesive systems were more technique sensitive and needed closer attention to application technique to achieve maximum bond strength (Miyazaki et.al., 1996).

The application of bonding agents, using manufacturer's instructions and simulated application errors of third-, fourth-, and fifth-generation systems was investigated in 2000. Simulated mistakes included prolonged etching, excessive drying, drying primers prematurely and drying primers excessively. The excess drying of primer could be compensated for by the application of a second coat. It was concluded that adherence to manufacturer's instructions were essential to maintain bond strengths and that the simplified materials were not less technique sensitive. They also concluded that the main reason for the commercial success of simplified adhesives is the easy handling, convenience, and the perception that there is less complexity of application steps when compared to the older products. (Frankenberger and Kramer, 2000).

One technique parameter studied was the application technique (i.e., passive versus active application, with or without scrubbing) and how this may affect bond strengths.

Some of the studies have shown that there is notable enhancement of bond strength for bonding adhesives by varying the application activity. Other studies emphasized multiple layers of primer application, while some stressed drying between primer applications and other variable applications of light curing. There was discussion about how the variation of application techniques may have affected the hybrid layer in the dentin and the depth of penetration into dentin. The studies revealed that no matter how the modification of technique in the later generation materials progressed, the three-step, etch-and-rinse adhesive systems and the two-step, self-etch systems consistently out-performed the two-step, etch and rinse and the one-step self-etching adhesives. The authors concluded that etch and rinse adhesive systems still represented the dominant method to achieve high bond strengths (Frankenberger and Perdigao, 2001).

The influence of deviations from the manufacturer's instructions for the use of six adhesive systems on the bond strengths to enamel and dentin was investigated in 2002. The investigators surveyed dentists on their use of adhesive systems with 21 systems reported, but only 6 systems were used by a substantial number of reporting dentists. A list of common clinical procedures was provided and dentists responded from memory using this list to guide their response to identify the order and durations used in their method of placement of the adhesive systems. Gluma Classic, Gluma CPs (Heraeus), Prime & Bond 2.1 (Dentsply), Scotchbond 1 and Scotchbond Multipurpose (3M-ESPE) and Syntac (Ivoclar, Amherst, NY) were used with various

composite materials. The adhesives were applied according to manufacturer's directions and then by the deviations identified in the survey of practitioner's techniques. The questionnaires identified deviations from manufacturer's instructions that were generally of the same type. Most deviations reported were to save time or simplify the procedures. Variations in technique for Scotchbond 1 and Scotchbond Multipurpose were found to affect dentin bond strengths, but enamel bond strengths remain unchanged. Overall, the study found that deviations from manufacturer's protocols significantly affected the bond of three of the six systems tested. They made particular note that ScotchBond Multipurpose showed particular technique sensitivity with regard to bonding strength to dentin (Peutzfeldt and Asmussen, 2002).

The effect of dentin bonding agent interaction and clinical application techniques on the shear bond strength of four dental adhesives: Optibond FL (three-step, etch-and-rinse), Clearfil SE Bond (two-step, self-etch), PQ1 (Ultradent) (three-step, etch-and-rinse) and Prime and Bond NT (two-step, etch-and-rinse) were tested in 2003. Application was varied by air spreading excess adhesive resins, or by clean brush removal of excess adhesive resins. Higher bond strengths were detected with one-second air removal (Bonilla and Stevenson, 2003).

In 2006, it was claimed that agitation of three self etch bonding agents at three different application times did not consistently improve shear bond strength to dentin for two-step, self-etching Clearfil SE Bond, AdhesSE (Ivoclar) and the one step Xeno

III (Dentsply). The primer was agitated for various time intervals for ClearFil SE Bond and AdheSE, while the Xeno III was applied in a single step and tested with or without agitation. They found differences between products for dentin bond strengths. Twenty seconds of agitation improved shear bond strength to dentin for all systems tested, while ten seconds improved dentin bond strength for Clearfil SE, while thirty seconds of agitation had no effect on any dentin bond strengths (Velasquez and Sergent 2006).

The effects of the degree of moisture and rubbing action on immediate dentin bond strengths of fifth generation, two-step, etch-and-rinse ethanol/water-based and acetone-based adhesive systems was tested in 2006. The finding was that light or vigorous rubbing action of ethanol/water and acetone-based adhesives is essential to provide high immediate bond strength to dentin (Dal-Bianco et.al, 2006).

Active and passive (rubbing, no rubbing) applications were compared in 2009 using the self-etching adhesives One Up Bond F (Tokuyama, Tokyo, Japan), Clearfil SE and Self & Etch (Vigodent, Rio de Janeiro, Brazil). They were compared alongside a "conventional adhesive" Magic Bond (Vigodent, Rio de Janeiro Brazil) which was used as a control. The authors reported that the active application of two layers of the self-etching adhesive systems produced significant improvement in bond strength to enamel compared to the passive application of one layer. (Torres and Barcellos, 2009)

The effect of application mode on micro-tensile bond strength of three, one-step self-adhesive systems (Clearfil S3 (Kuraray), Xeno III, and Adper Prompt-L Pop) was studied again in 2010. The investigators concluded that application with agitation on dentin improves bond strength of one-step self-etch adhesives (Amaral and Stanislawczuk, 2010).

Soares and Carracho (2010) evaluated the relationship between the number of adhesive layers and internal adaptation on the microtensile bond strength to enamel and dentin. Scotchbond Multipurpose (three-step, etch-and-rinse), Adper Single Bond 2 (3M-ESPE) (one-step, self-etch) and Clearfil SE Bond (two-step, self-etch) were evaluated. Two layer application of the adhesive with light curing after each application showed slight increases in bond strength and improvement in internal adaption, but the formation of internal failures in restorations was not minimized. The three-step etch-and-rinse adhesive, Scotchbond Multipurpose, showed the best performance over simplified adhesives. Low tensile bond strengths correlated to higher numbers of cracks and ruptures.

Taken together as a whole, these studies hint at the possibility that the performance of three-step, etch-and-rinse adhesives may be altered by changing application technique when bonding to dentin. The Miyazaki study looked at active versus passive application of the primer component, but in a very limited way. None of the studies specifically investigated the relationship between the variable of active versus passive

application of the primer component as a separate issue alone, or compared variances in time, when evaluating the bond strength of three-step, etch-and-rinse adhesives to dentin.

The review of the literature led to the conclusion that the purpose of this study should be to investigate the effect of application technique of the primer, particularly active or passive application, on the shear bond strength of fourth-generation, three-step, etch-and-rinse adhesive bonding agents to dentin. The question to be answered is whether their already excellent bond strength characteristics can be further enhanced by modification of application technique.

Affirmative results could solidify the rational for preferential use of this class of materials in clinical practice as the benefits of improved bond strength and demonstrated longer term restorative success could outweigh the disadvantages of their slightly more complicated application protocols.

II. OBJECTIVES

A. Objectives Overview

The purpose of this study was to evaluate the effect of variations in application technique of the primer component of three-step, etch-and-rinse adhesive agents on the shear bond strength of composite resin to dentin.

B. Specific Hypotheses

a. <u>Null Hypotheses:</u>

There is no significant difference in the shear bond strength of composite to dentin using Optibond FL based on primer application: 1) time or 2) method.

b. <u>Null Hypotheses:</u>

There is no significant difference in the shear bond strength of composite to dentin using Adper Scotchbond Multipurpose based on primer application: 1) time, or 2) method.

III. MATERIALS AND METHODS

A. Experimental Design

One hundred twenty extracted human third molars were stored in 0.5% chloramine at 4° C. The molars were used within 6 months following extraction. Teeth were mounted in dental stone inside a section of PVC pipe with the crown exposed and accessible. A diamond saw (Isomet, Buehler, Lake Forest, IL) was used to remove 2mm or more coronal tooth structure to ensure dentin exposure and the proper orientation of the surface relative to the direction of shear force applied. Each specimen was then examined under a stereomicroscope (SMZ-1B, Nikon, Melville, NY) at 10X magnification to ensure complete exposure of the dentin surface with no residual enamel. A uniform smear layer was created on the flat dentin surfaces using two passes on 600-grit carbide paper. See Figure 1 (A-D).

The mounted specimens were divided into two groups of bonding agents. The materials evaluated were Scotchbond Multipurpose and Optibond FL. All surface conditioning steps were performed according to manufacturer's instructions. The dentin surface was etched and rinsed using the product specific phosphoric-acid gel following the manufacturer's directions. The appropriate adhesive primers were applied with passive application or active scrubbing with three various time applications: 1)manufacturer's recommended application time, 2) manufacturer's recommended application time plus 10 seconds, manufacturer's recommended

application time plus 20 seconds) using similar brand and shaped applicators. Manufacturer's directions were followed with regards to intermediate application steps (i.e., air drying, number of applications) with the only variables and deviations from application instructions being a) active or passive application and b) application time. See Figure 1 (E-G), Figure 2 and Table 2.

The adhesives were cured as recommended by the manufacturer using the Bluephase 16i (Ivoclar) light-curing unit. Irradiance of the curing light was determined with a radiometer (LED Radiometer, Kerr) to verify irradiance levels above 1200 mW/cm^2 and was re-verified for each group of ten samples.

The specimens were placed in an Ultradent Jig and secured beneath a white non-stick Delrin insert (Ultradent, South Jordan, UT). The resin composite (Filtek Z250, Shade A2, 3M ESPE) was applied in 2-mm incremental layers to a height of 3-4mm. The bonding area was limited to a 2.4mm diameter circle determined by the Delrin insert. Each layer was light cured for 20 seconds as recommended by the manufacturers. All specimens were stored 24 hours in distilled water at 37^{0}C. See Figure 3.

The samples were then loaded perpendicularly with a customized probe (Ultradent) in a universal testing machine (Instron) using a crosshead speed of 1.0 mm/min until bonding failure occurred. See Figure 4.

Shear bond strength values in megapascals (MPa) were calculated from the peak load of failure (newtons) divided by the specimen surface area. The mean and standard deviation were determined for each group.

Following testing, each specimen was examined using 10X stereomicroscope to determine failure mode as either: a) adhesive fracture at the composite/adhesive/dentin interface; b) cohesive fracture in composite; c) mixed (combined adhesive and cohesive) in the composite and bonded interface, or between dentin and bonded interface; d) cohesive fracture in dentin.
See Figure 5.

Figure 1.   Specimen Preparation

A - Sample preparation material

B- Isomet diamond saw

C- Mounted after diamond saw cut

D- Mounted in PVC ring

E-   Etching sample
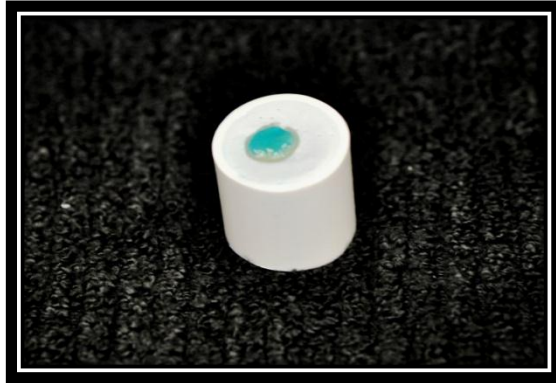
F-   Passive brush illustration- straight handle without scrubbing.

G-   Active brush illustration- bent handle with scrubbing.

Figure 2. **Experimental Groupings**

- OPTIBOND FL (OFL) (n=60)
  - ACTIVE APPLICATION (n=30)
    - MANUFACTURER'S DIRECTIONS (MD) ACTIVE OFL (n=10)
    - MD+10 ACTIVE OFL (n=10)
    - MD+20 ACTIVE OFL (n=10)
  - PASSIVE APPLICATION (n=30)
    - MANUFACTURER'S DIRECTIONS (MD) PASSIVE OFL (n=10)
    - MD+10 PASSIVE OFL (n=10)
    - MD+20 PASSIVE OFL (n=10)
- SCOTCHBOND MULTIPURPOSE (SBMP) (n=60)
  - ACTIVE APPLICATION (n=30)
    - MANUFACTURER'S DIRECTIONS (MD) ACTIVE SBMP (n=10)
    - MD+10 ACTIVE SBMP (n=10)
    - MD+20 ACTIVE SBMP (n=10)
  - PASSIVE APPLICATION (n=30)
    - MANUFACTURER'S DIRECTIONS (MD) PASSIVE SBMP (n=10)
    - MD+10 PASSIVE SBMP (n=10)
    - MD+20 PASSIVE SBMP (n=10)

TABLE 2.

Manufacturer's Directions (MD) for each material, and time variances of 10 and 20 seconds from MD with active and passive application.

	STEP 1 MD	STEP 2 MD	STEP 3 MD	STEP 3 MD+10s	STEP 3 MD+20s	STEP 4 MD	STEP 5 MD
Optibond FL (Kerr)	**Etch:** Kerr Gel Etchant 35% Phosphoric Acid 15 seconds	**Rinse:** Thoroughly for 15 secs; Followed by: air dry for 3 seconds Do not dessicate	**Prime:** Apply with light brushing motion for 15 secs; Air dry for 5 seconds	**Prime:** **(P)assive:** light brushing motion 25 seconds **(A)ctive** scrub motion 25 seconds Air dry 5 seconds	**Prime:** **(P)assive:** light brushing motion 35 seconds **(A)ctive** scrub 35 seconds Air dry 5 seconds	**Adhesive:** Using same applicator apply with light brushing motion for 15 seconds Air thin 3 seconds	**Light cure:** 20 seconds
Scotchbond Multi-Purpose (3M-ESPE)	**Etch:** Scotchbond Etchant (phosphoric or maleic) Wait 15 seconds	**Rinse:** Rinse 15 seconds Air dry 5 seconds Leave moist	**Prime:** Apply: Dry gently for 5 seconds	**Prime:** Apply: **(P)assive:** Dwell 10 secs **(A)ctive:** scrub 10 secs, Dry gently: 5 seconds	**Prime:** Apply: **(P)assive:** Dwell 20 seconds **(A)ctive:** scrub 20 secs, Dry gently: 5 seconds	**Adhesive:** Apply	**Light cure:** 10 seconds

Figure 3   Sample fabrication using Ultradent jig

A- 2mm composite increment
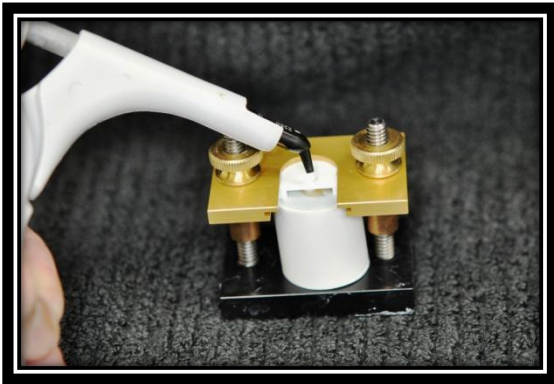
B- Condenser used for increments
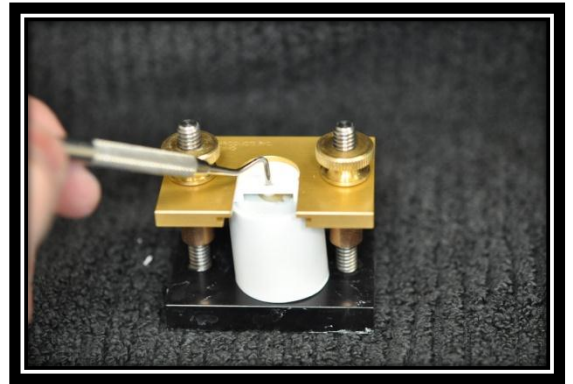
C- Application of curing light
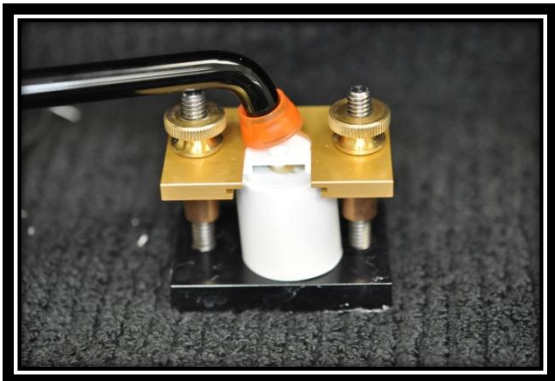
D- Sample ready for shear testing
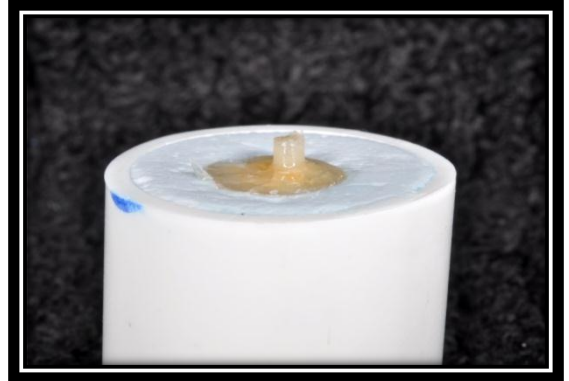
Figure 4   MTBS Shear Bond Strength Test

A-   Loading samples on Instron universal testing machine
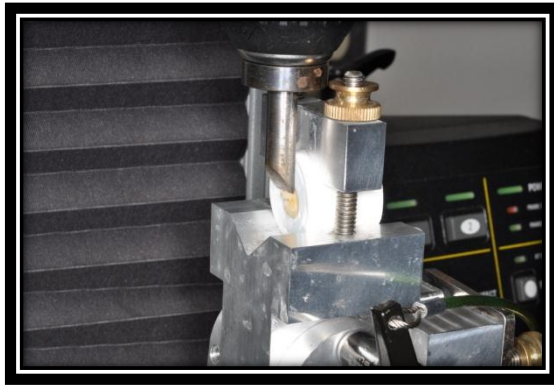
Figure 5   Analysis of Fracture Mode (representative SEM images)

A.   Mixed Fracture (composite – adhesive interface)
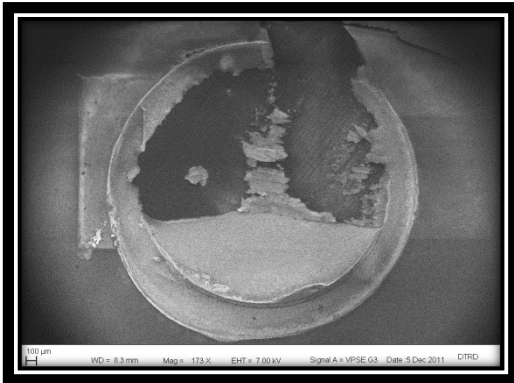
B.   Adhesive Fracture (adhesive interface)
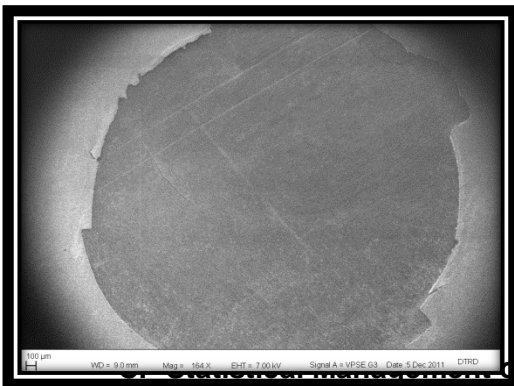
B. Statistical Management of Data

1. Data Analysis:

Shear bond strength data was analyzed with a three-way ANOVA and Tukey's post-hoc tests to evaluate the effects of bonding agent (2 levels), primer treatment time (3 levels), and primer action (2 levels) on the shear bond strength of composite to dentin (alpha = 0.05).

2. Sample size estimation/power analysis

The sample size of 10 per group provided 80% power to detect the following effect size differences: 0.258 (or approximately 0.56 standard deviation between the means) for the main effects of bonding agent (2 levels) and primer action (2 levels) and 0.288 (or approximately 0.58 standard deviation among the means) for the main effect of primer treatment time (3 levels) with interaction effect sizes ranging from 0.258 to 0.287 when testing with a 3-factor ANOVA at the alpha level of 0.05 (NCSS PASS 2002). See Table 3.

TABLE 3.   Sample size estimation/power analysis

Term	Power n	n	Total N	df1	df2	Std Dev of Means(Sm)	Size	Effect Alpha	Beta
A	0.79832	10.00	120	1	108	0.258	0.258	0.05000	0.20168
B	0.80066	10.00	120	2	108	0.288	0.288	0.05000	0.19934
C	0.79832	10.00	120	1	108	0.258	0.258	0.05000	0.20168
AB	0.79892	10.00	120	2	108	0.287	0.287	0.05000	0.20108
AC	0.79876	10.00	120	1	108	0.258	0.258	0.05000	0.20124
BC	0.79892	10.00	120	2	108	0.287	0.287	0.05000	0.20108

IV. RESULTS

The three-way ANOVA found significant differences in the shear bond strength of the composite to dentin based on application time (p=0.041), but not on application method (p=0.051) or bonding agent (p=0.059). However, there was a significant interaction with application time and method (p=0.001). Further statistical analysis was completed using a two-way ANOVA per bonding agent type.

With Optibond FL, a two-way ANOVA found no significant difference between groups based on application time or method, with no significant interaction (p>0.05).

With Scotchbond Multipurpose, a two-way ANOVA found no significant differences based on application time or method (p>0.05), but there was a significant interaction (p=0.002).

Further statistical analysis with one-way ANOVAs and t-tests for Adper Scotchbond Multipurpose found a significant difference between groups (p<0.01). A Bonferroni correction with an alpha level of 0.017 was applied because several statistical tests were performed simultaneously. See Table 4 and Figure 6.

When examining fracture mode, Adper Scotchbond Multipurpose had primarily mixed failures at the adhesive/composite interface, whereas Optibond FL had a more even distribution between adhesive and mixed failures. See Figure 7.

Table 4   Summary of Mean Shear Bond Strength and Standard Deviations

Application Time (seconds) Manufacturer's Directions (MD)	Mean Shear Bond Strength MPa (st dev)			
	Scotchbond MP		Optibond FL	
	Active	Passive	Active	Passive
MD	17.46 (6.33) Aa	8.60 (4.66) Ba	14.27 (4.91)	9.92 (6.20)
MD + 10 secs	11.19 (6.48) Ab	11.61 (2.91) Aa	10.20 (5.39)	7.80 (4.25)
MD + 20 secs	9.21 (3.36) Ab	12.57 (6.53) Aa	8.50 (3.64)	9.33 (4.49)
	Groups with the same lower case letter per column or upper case letter by row are not significantly different (p>0.05)			

Figure 6 – Shear Bond Strength

MPa

■ Manufacturer's Directions   ■ Manufacturer's Directions +10s   ■ Manufacturer's Directions +20s
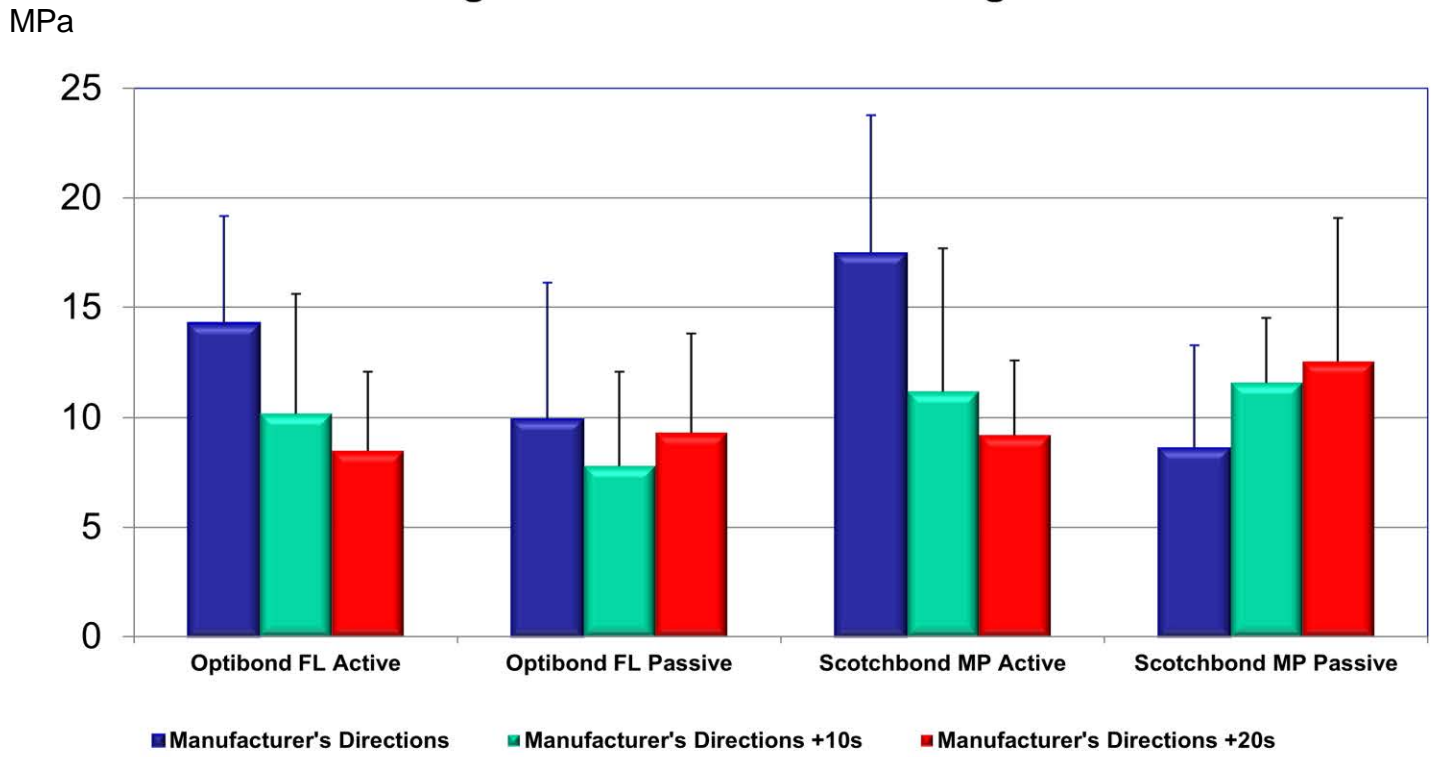
Figure 7 - Fracture Mode
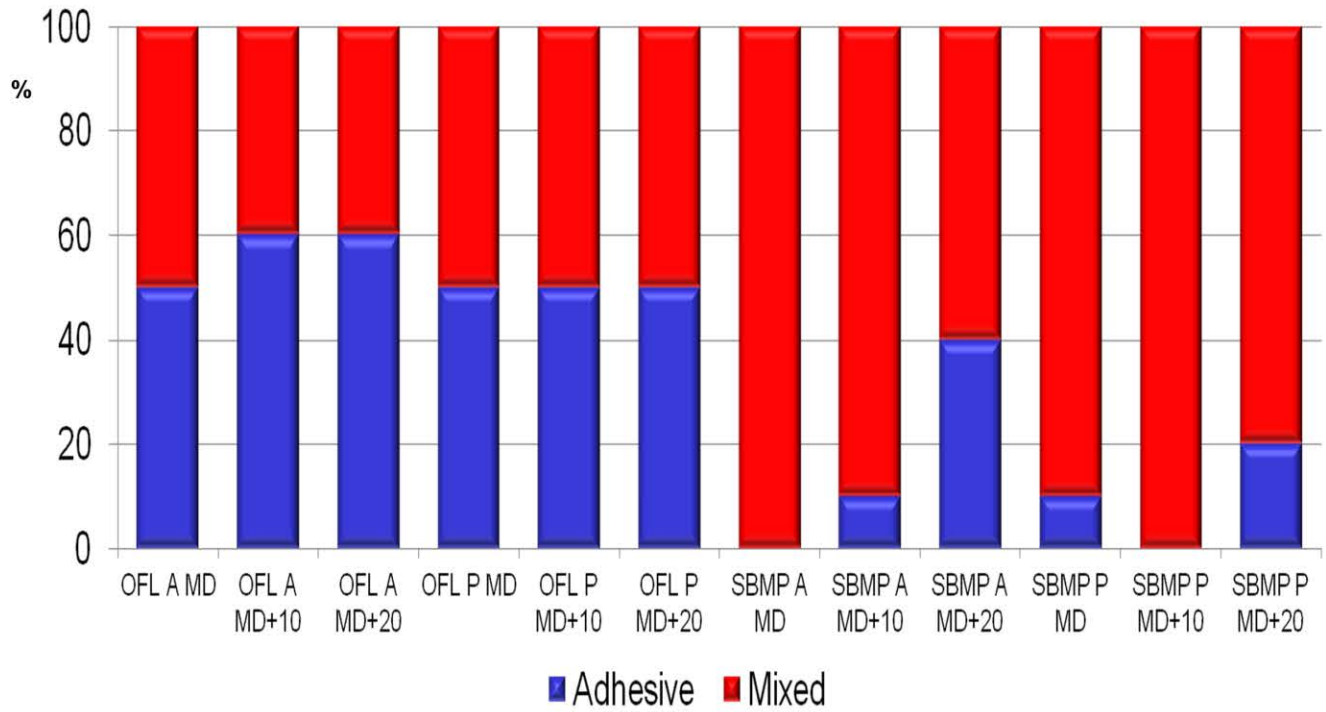
V. DISCUSSION

The assumptions made as this investigation began were that there would be no difference in shear bond strength between specimens of OptiBond FL or Adper Scotchbond Multipurpose based on changes in application time or application technique of the primer. The manufacturer's directions as written are fairly specific with regards to time of etching, rinsing, air drying, and adhesive application.

Notable in the manufacturer's directions for application of the primer component of Optibond FL is the language to "apply with a light brushing motion for 15 seconds", whereas the language in the manufacturer's directions for the primer component application of Adper Scotchbond Multipurpose simply states "apply", with no reference to a specific application technique or time.

We failed to reject the first null hypothesis. No significant difference in the shear bond strength of composite to dentin was found using Optibond FL based on primer application time or method.

The second null hypothesis was rejected. A significant difference in the shear bond strength of composite to dentin was found using Adper Scotchbond Multipurpose. However, the differences were technique specific. Active primer application was only significantly different from passive application at the manufacturer's suggested application time and application time was only significantly different with active

application of the primer. As previously noted, the Miyazaki study reported that the fourth-generation adhesive systems were more technique sensitive and needed closer attention to application technique in order to achieve maximum bond strength. (Miyazaki et.al., 1996).

Peutzfeldt demonstrated that adherence to manufacturer's directions was very important to maximize bond strengths to dentin and that deviations in technique could adversely affect bond strength in multiple systems, particularly Scotchbond Multipurpose. (Peutzfeldt and Asmussen, 2002).

Based on the results of this investigation, there is no demonstrable statistical basis for varying from the manufacturer's directions for Optibond FL. It is a significant finding that the active application of the primer of Adper Scotchbond Multipurpose resulted in an increase in bond strengths only at the manufacturer's directed time of application. This same effect does not occur when longer application times or passive application of the primer component of Adper Scotchbond Multipurpose are used.

The three-way ANOVA found no significant difference between the two bonding agents. However, Optibond FL had more adhesive failures than Adper Scotchbond Multipurpose, suggesting a weaker adhesive interface.

This investigation, along with the studies previously cited in the literature taken as a whole, provide more evidence that confirms the technique sensitivity of the application of Adper Scotchbond Multipurpose. Also, it does not seem unreasonable to suggest an alteration to the manufacturer's directions for Adper Scotchbond Multipurpose that would be to include additional specific language that directs an "active" application technique. That modification, substantiated by the evidence provided in this study, will increase the likelihood of improving the excellent characteristics of this "gold standard" adhesive bonding system.

Clinical judgment concerning specific application situations of these adhesive systems must always continue to guide the clinician. Deciding between the less complicated and less time consuming fifth-, sixth- and seventh-generation bonding materials, or the generally stronger fourth-generation adhesive systems and their particular performance characteristics will depend on specific clinical and treatment requirements. The higher bond strengths of fourth-generation, etch-and-rinse adhesives, particularly Adper Scotchbond Multipurpose with active application, may improve the clinician's probability of long-term restorative success. Thus, this class of adhesives would be a preferable selection in clinical scenario that requires high bond strength and longer restoration retention.

VI.   CONCLUSIONS

A.   With Optibond FL, there was no significant difference in shear bond strength of composite to dentin based on application time, or active versus passive application of the primer.

B.   Active application of the primer of Adper Scotchbond Multipurpose at the manufacturer's recommended application time increased the bond strength compared to longer application times or passive application.

Appendix A: Scotchbond Multipurpose Shear Bond Strength Raw data values

Fx=Fracture mode: (A) Adhesive, (MC) Mixed Composite

Sample	MD Active N	MPa	Fx Mode	MD Passive N	MPa	Fx Mode
1	99.465	22.55	MC	59.084	13.40	MC
2	62.476	14.17	MC	56.116	12.72	MC
3	62.767	14.23	MC	10.684	2.42	MC
4	78.125	17.72	MC	29.624	6.72	MC
5	86.463	19.61	MC	61.372	13.92	A
6	28.232	6.40	MC	22.488	5.10	MC
7	101.357	22.98	MC	52.911	12.00	MC
8	119.919	27.19	MC	54.148	12.28	MC
9	86.990	19.73	MC	13.816	3.13	MC
10	44.031	9.98	MC	19.142	4.34	MC
	avg	17.46		avg	8.60	
	st dev	6.33		st dev	4.66	
Sample	MD + 10 Active N	MPa	Fx Mode	MD + 10 Passive N	MPa	Fx Mode
1	101.671	23.05	MC	74.149	16.81	MC
2	56.689	12.85	MC	42.989	9.75	MC
3	44.778	10.15	MC	48.519	11.00	MC
4	34.409	7.80	MC	52.838	11.98	MC
5	17.018	3.86	MC	30.228	6.85	MC
6	66.344	15.04	MC	53.397	12.11	MC
7	25.972	5.89	MC	56.218	12.75	MC
8	17.890	4.06	MC	37.514	8.51	MC
9	42.133	9.55	MC	50.224	11.39	A
10	86.735	19.67	A	65.955	14.96	MC
	avg	11.19		avg	11.61	
	st dev	6.48		st dev	2.91	
Sample	MD + 20 Active N	MPa	Fx Mode	MD + 20 Passive N	MPa	Fx Mode
1	67.102	15.22	MC	74.027	16.79	MC
2	46.546	10.55	A	15.127	3.43	MC
3	46.511	10.55	MC	96.091	21.79	MC
4	22.829	5.18	A	75.851	17.20	MC
5	19.168	4.35	A	65.884	14.94	A
6	58.632	13.30	MC	87.067	19.74	MC
7	38.496	8.73	A	43.519	9.87	MC
8	34.804	7.89	MC	47.343	10.74	A
9	32.486	7.37	MC	35.580	8.07	MC
10	39.572	8.97	MC	13.993	3.17	MC
	avg	9.21		avg	12.57	
	st dev	3.36		st dev	6.53	

Appendix B: Optibond FL Shear Bond Strength Raw data values

Fx=Fracture mode: (A) Adhesive, (MC) Mixed-Composite

Sample	MD Active N	MPa	Fx Mode	MD Passive N	MPa	Fx Mode
	Optibond FL					
	MD **Active**			**MD** **Passive**		
Sample	**N**	**MPa**	**Fx Mode**	**N**	**MPa**	**Fx Mode**
1	44.247	10.03	MC	24.720	5.61	MC
2	55.299	12.54	A	38.410	8.71	A
3	65.036	14.75	A	6.804	1.54	MC
4	29.428	6.67	MC	48.231	10.94	A
5	78.044	17.70	MC	54.457	12.35	A
6	48.138	10.92	A	90.806	20.59	MC
7	92.534	20.98	A	70.303	15.94	MC
8	62.529	14.18	MC	18.206	4.13	A
9	99.031	22.46	A	17.551	3.98	MC
10	55.106	12.50	MC	68.000	15.42	A
	avg	**14.27**		**avg**	**9.92**	
	st dev	**4.91**		**st dev**	**6.20**	
	MD + 10 **Active**			**MD + 10** **Passive**		
Sample	**N**	**MPa**	**Fx Mode**	**N**	**MPa**	**Fx Mode**
1	53.451	12.12	A	52.999	12.02	MC
2	37.139	8.42	MC	15.052	3.41	A
3	48.237	10.94	A	23.323	5.29	A
4	30.344	6.88	MC	50.093	11.36	A
5	31.254	7.09	A	20.937	4.75	MC
6	65.853	14.93	MC	12.237	2.77	MC
7	25.860	5.86	A	60.293	13.67	A
8	18.056	4.09	A	28.514	6.47	MC
9	40.044	9.08	MC	57.984	13.15	A
10	99.655	22.60	A	22.374	5.07	MC
	avg	**10.20**		**avg**	**7.80**	
	st dev	**5.39**		**st dev**	**4.25**	
	MD + 20 **Active**			**MD + 20** **Passive**		
Sample	**N**	**MPa**	**Fx Mode**	**N**	**MPa**	**Fx Mode**
1	61.337	13.91	A	79.320	17.99	A
2	33.144	7.52	A	39.396	8.93	MC
3	57.980	13.15	MC	29.762	6.75	MC
4	37.880	8.59	MC	9.291	2.11	A
5	19.800	4.49	A	33.142	7.52	MC
6	33.484	7.59	A	28.183	6.39	A
7	12.430	2.82	MC	49.164	11.15	A
8	51.126	11.59	A	55.695	12.63	A
9	41.976	9.52	A	29.675	6.73	MC
10	25.525	5.79	MC	58.020	13.16	MC
	avg	**8.50**		**avg**	**9.33**	
	st dev	**3.64**		**st dev**	**4.49**	

Appendix C: 3-way ANOVA

Notes		
Output Created		02-AUG-2011 07:27:56
Comments		
Input	Data	E:\Zald\3-way anova.sav
	Filter	<none>
	Weight	<none>
	Split File	<none>
	N of Rows in Working Data File	120
Missing Value Handling	Definition of Missing	User-defined missing values are treated as missing.
	Cases Used	Statistics are based on all cases with valid data for all variables in the model.
Syntax		UNIANOVA mpa BY time action agent /METHOD = SSTYPE(3) /INTERCEPT = INCLUDE /POSTHOC = time (TUKEY) /PRINT = DESCRIPTIVE /CRITERIA = ALPHA(.05) /DESIGN = time action agent time*action time*agent action*agent time*action *agent .
Resources	Elapsed Time	0:00:00.34

3-way ANOVA

TIME	ACTION	AGENT	Mean	Std. Deviation	N
			Descriptive Statistics		
			Dependent Variable: MPA		
ten	**act**	**op**	10.2010	5.3919	10
		sb	11.1920	6.4813	10
		Total	10.6965	5.8248	20
	pas	**op**	7.7960	4.2552	10
		sb	11.6110	2.9078	10
		Total	9.7035	4.0512	20
	Total	**op**	8.9985	4.8857	20
		sb	11.4015	4.8938	20
		Total	10.2000	4.9777	40
twen	**act**	**op**	8.4970	3.6357	10
		sb	9.2110	3.3618	10
		Total	8.8540	3.4277	20
	pas	**op**	9.3360	4.4906	10
		sb	12.5740	6.5325	10
		Total	10.9550	5.7031	20
	Total	**op**	8.9165	3.9998	20
		sb	10.8925	5.3426	20
		Total	9.9045	4.7646	40
zero	**act**	**op**	14.2730	4.9124	10
		sb	17.4560	6.3283	10
		Total	15.8645	5.7504	20
	pas	**op**	9.9210	6.2036	10
		sb	8.6030	4.6603	10
		Total	9.2620	5.3828	20
	Total	**op**	12.0970	5.8860	20
		sb	13.0295	7.0628	20
		Total	12.5633	6.4345	40
Total	**act**	**op**	10.9903	5.1661	30
		sb	12.6197	6.4612	30
		Total	11.8050	5.8577	60
	pas	**op**	9.0177	4.9651	30
		sb	10.9293	5.0564	30
		Total	9.9735	5.0609	60
	Total	**op**	10.0040	5.1210	60
		sb	11.7745	5.8149	60
		Total	10.8892	5.5278	120

3-way ANOVA

Tests of Between-Subjects Effects Dependent Variable: MPA					
Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	851.812(a)	11	77.437	3.004	.002
Intercept	14229.092	1	14229.092	551.902	.000
TIME	169.883	2	84.941	3.295	.041
ACTION	100.632	1	100.632	3.903	.051
AGENT	94.040	1	94.040	3.648	.059
TIME * ACTION	389.301	2	194.650	7.550	.001
TIME * AGENT	11.445	2	5.723	.222	.801
ACTION * AGENT	.598	1	.598	.023	.879
TIME * ACTION * AGENT	85.914	2	42.957	1.666	.194
Error	2784.447	108	25.782		
Total	17865.351	120			
Corrected Total	3636.260	119			
a R Squared = .234 (Adjusted R Squared = .156)					

Appendix D: 2-way ANOVA SBMP

Notes		
Output Created		02-AUG-2011 07:11:44
Comments		
Input	**Data**	E:\Zald\2-way anova SB.sav
	Filter	<none>
	Weight	<none>
	Split File	<none>
	N of Rows in Working Data File	60
Missing Value Handling	**Definition of Missing**	User-defined missing values are treated as missing.
	Cases Used	Statistics are based on all cases with valid data for all variables in the model.
Syntax		UNIANOVA mpa BY time action /METHOD = SSTYPE(3) /INTERCEPT = INCLUDE /POSTHOC = time action (TUKEY) /PRINT = DESCRIPTIVE /CRITERIA = ALPHA(.05) /DESIGN = time action time*action .
Resources	**Elapsed Time**	0:00:03.39

Descriptive Statistics
Dependent Variable: MPA

TIME	ACTION	Mean	Std. Deviation	N
ten	act	11.1920	6.4813	10
	pas	11.6110	2.9078	10
	Total	11.4015	4.8938	20
twen	act	9.2110	3.3618	10
	pas	12.5740	6.5325	10
	Total	10.8925	5.3426	20
zero	act	17.4560	6.3283	10
	pas	8.6030	4.6603	10
	Total	13.0295	7.0628	20
Total	act	12.6197	6.4612	30
	pas	10.9293	5.0564	30
	Total	11.7745	5.8149	60

Tests of Between-Subjects Effects
Dependent Variable: MPA

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	499.146(a)	5	99.829	3.604	.007
Intercept	8318.331	1	8318.331	300.293	.000
TIME	49.842	2	24.921	.900	.413
ACTION	42.858	1	42.858	1.547	.219
TIME * ACTION	406.446	2	203.223	7.336	.002
Error	1495.837	54	27.701		
Total	10313.315	60			
Corrected Total	1994.983	59			
a R Squared = .250 (Adjusted R Squared = .181)					

Appendix E: 2-way ANOVA Optibond FL

Notes		
Output Created		25-JUL-2011 06:44:20
Comments		
Input	**Data**	E:\Zald\2-way anova Opti.sav
	Filter	\<none\>
	Weight	\<none\>
	Split File	\<none\>
	N of Rows in Working Data File	60
Missing Value Handling	**Definition of Missing**	User-defined missing values are treated as missing.
	Cases Used	Statistics are based on all cases with valid data for all variables in the model.

Syntax		UNIANOVA
		    mpa BY time action
		    /METHOD = SSTYPE(3)
		    /INTERCEPT = INCLUDE
		    /POSTHOC = time action (TUKEY)
		    /PRINT = DESCRIPTIVE
		    /CRITERIA = ALPHA(.05)
		    /DESIGN = time action time*action .

Resources	**Elapsed Time**	0:00:01.18

Descriptive Statistics
Dependent Variable: MPA

TIME	ACTION	Mean	Std. Deviation	N
ten	act	10.2010	5.3919	10
	pas	7.7960	4.2552	10
	Total	8.9985	4.8857	20
twen	act	8.4970	3.6357	10
	pas	9.3360	4.4906	10
	Total	8.9165	3.9998	20
zero	act	14.2730	4.9124	10
	pas	9.9210	6.2036	10
	Total	12.0970	5.8860	20
Total	act	10.9903	5.1661	30
	pas	9.0177	4.9651	30
	Total	10.0040	5.1210	60

Tests of Between-Subjects Effects
Dependent Variable: MPA

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	258.626(a)	5	51.725	2.168	.071
Intercept	6004.801	1	6004.801	251.635	.000
TIME	131.487	2	65.743	2.755	.073
ACTION	58.371	1	58.371	2.446	.124
TIME * ACTION	68.768	2	34.384	1.441	.246
Error	1288.610	54	23.863		
Total	7552.037	60			
Corrected Total	1547.236	59			

a R Squared = .167 (Adjusted R Squared = .090)

Appendix F:　　1-way ANOVA SBMP Passive

Notes		
Output Created		02-AUG-2011 07:19:10
Comments		
Input	**Data**	E:\Zald\1-way anova SB pass.sav
	Filter	<none>
	Weight	<none>
	Split File	<none>
	N of Rows in Working Data File	30
Missing Value Handling	**Definition of Missing**	User-defined missing values are treated as missing.
	Cases Used	Statistics are based on all cases with valid data for all variables in the model.
Syntax		UNIANOVA 　mpa　　　　　　　　　　BY　　　　　　　　time 　/METHOD　　　　　　=　　　　　SSTYPE(3) 　/INTERCEPT　　　　　=　　　　　INCLUDE 　/POSTHOC　　=　　time　　(　　TUKEY　　) 　/PRINT　　　　　　　=　　　　DESCRIPTIVE 　/CRITERIA　　　　　=　　　　ALPHA(.05) 　/DESIGN = time .
Resources	**Elapsed Time**	0:00:00.27

Descriptive Statistics
Dependent Variable:
MPA

TIME	Mean	Std. Deviation	N
ten	11.6110	2.9078	10
twen	12.5740	6.5325	10
zero	8.6030	4.6603	10
Total	10.9293	5.0564	30

Tests of Between-Subjects Effects
Dependent Variable: MPA

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	85.814(a)	2	42.907	1.767	.190
Intercept	3583.510	1	3583.510	147.575	.000
TIME	85.814	2	42.907	1.767	.190
Error	655.629	27	24.283		
Total	4324.953	30			
Corrected Total	741.443	29			
a R Squared = .116 (Adjusted R Squared = .050)					

Appendix G: 1-way ANOVA SBMP Active

Notes		
Output Created		02-AUG-2011 07:15:45
Comments		
Input	**Data**	E:\Zald\1-way anova SB active.sav
	Filter	<none>
	Weight	<none>
	Split File	<none>
	N of Rows in Working Data File	30
Missing Value Handling	**Definition of Missing**	User-defined missing values are treated as missing.
	Cases Used	Statistics are based on all cases with valid data for all variables in the model.
Syntax		UNIANOVA mpa BY time /METHOD = SSTYPE(3) /INTERCEPT = INCLUDE /POSTHOC = time (TUKEY) /PRINT = DESCRIPTIVE /CRITERIA = ALPHA(.05) /DESIGN = time .
Resources	**Elapsed Time**	0:00:00.47

Descriptive Statistics
Dependent Variable:
MPA

TIME	Mean	Std. Deviation	N
ten	11.1920	6.4813	10
twen	9.2110	3.3618	10
zero	17.4560	6.3283	10
Total	12.6197	6.4612	30

Tests of Between-Subjects Effects
Dependent Variable: MPA

Source	Type III Sum of Squares	df	Mean Square	F	Sig.
Corrected Model	370.474(a)	2	185.237	5.953	.007
Intercept	4777.680	1	4777.680	153.530	.000
TIME	370.474	2	185.237	5.953	.007
Error	840.208	27	31.119		
Total	5988.362	30			
Corrected Total	1210.682	29			
a R Squared = .306 (Adjusted R Squared = .255)					

Post Hoc Tests

TIME

					95% Confidence Interval	
(I) TIME	(J) TIME	Mean Difference (I-J)	Std. Error	Sig.	Lower Bound	Upper Bound
ten	twen	1.9810	2.4947	.710	-4.2045	8.1665
	zero	-6.2640(*)	2.4947	.047	-12.4495	-7.8466E-02
twen	ten	-1.9810	2.4947	.710	-8.1665	4.2045
	zero	-8.2450(*)	2.4947	.007	-14.4305	-2.0595
zero	ten	6.2640(*)	2.4947	.047	7.847E-02	12.4495
	twen	8.2450(*)	2.4947	.007	2.0595	14.4305

Multiple Comparisons. Dependent Variable: MPA. Tukey HSD.

Based on observed means.

* The mean difference is significant at the .05 level.

Homogeneous Subsets

		Subset	
TIME	N	1	2
twen	10	9.2110	
ten	10	11.1920	
zero	10		17.4560
Sig.		.710	1.000

MPA. Tukey HSD.

Means for groups in homogeneous subsets are displayed.
Based on Type III Sum of Squares
The error term is Mean Square(Error) = 31.119.

a Uses Harmonic Mean Sample Size = 10.000.

b Alpha = .05.

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